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Procedures for the Use of Lexan and Makrofol SSNTDs in the Detection of

Environmental Concentrations of ^{235}U and ^{239}Pu.

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MASTER

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Table of Contents

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iii

List of Figures

iv

Abstract

Solid State Nuclear Track Detectors are used to study a variety of atomic particles. Polycarbonate SSNTD is used to study environmental concentrations of ^{235}U and ^{239}Pu in human urine and feces through fission track analysis. The samples of interest are deposited upon a Lexan slide, covered with a piece of Makrofol and exposed to a neutron fluence of 1.1×10^{17}. The fissile isotopes in the sample fission and the resulting fission fragments pass through either the surface of the Lexan or the surface of the Makrofol. The positive Coulombic attraction of the ionized fission fragments causes the electrons of the polycarbonate lattice to move towards the path of these particles, resulting in the breakage of chemical bonds in the lattice. The detector is then chemically etched in 6.5 N KOH that preferentially dissolves the damaged polycarbonate left in the path of the fission fragment. The chemically etched fission tracks are permanent records of the path of the fission fragment. The etched fission tracks in Lexan are optically counted using a microscope and the fission tracks in Makrofol are counted using a Spark Chamber. The amount of fissile material in the original sample can be calculated from the number of fission tracks. This paper presents further details of procedures for etching fission tracks in Lexan and Makrofol and for operating a Spark Chamber to count etched fission tracks in Makrofol. The physics of fission track formation in dielectric detectors is also discussed, as well as the physics of the Spark Chamber.

1

Introduction

The study of atomic particle concentrations and behavior using Solid State Nuclear Track Detectors (SSNTDs) is a relatively new and dynamic area of science. If the SSNTD is properly prepared and treated, i.e., chemically etched, it can provide a permanent record of the quantity and characteristics of the particles which have encountered its surface. A review of the literature in the field of SSNTDs shows that there have been applications using SSNTDs to detect a variety of particles, including protons, alpha particles, fission fragments, and cosmic rays.

There is substantial interest in the detection of low or environmental levels of several types of particles. An area of special interest is the detection of environmental levels of ^{235}U and ^{239}Pu. This project paper has its base in a study by the University of Utah and the U. S. Department of Energy (DOE) to detect ^{235}U and ^{239}Pu in the human body. For this project, these isotopes are chemically extracted and separated from a sample of interest and deposited upon a slide of polycarbonate SSNTD. When the SSNTD is exposed to a neutron fluence, the ^{235}U or ^{239}Pu will fission, sending fission fragments into the surface of the SSNTD. These fission fragments will create tracks in the SSNTD along their paths called "fission tracks". If the SSNTD is chemically etched after exposure to the neutron flux, the defects caused by the fission fragments will become permanent holes or tracks in the polycarbonate. These fission tracks can then be counted to provide an accurate number of the fission events within the sample that was deposited on the SSNTD's surface. Using this calculated number of fission events, an accurate estimate of the amount of ^{235}U or ^{239}Pu in the original sample of interest can be found [10].

This project paper provides a complete set of procedures used to chemically etch Lexan and Makrofol polycarbonate SSNTDs for use in fission track analysis and to provide the procedure for using a Spark Gap Chamber for counting the fission tracks in Makrofol. The background material necessary for understanding the physics of fission track registration and etching, and for the physics of the Spark Gap Chamber will also be provided in this paper.

2

Fission Track Registration

Fission track analysis can be done using many different combinations of Solid State Nuclear Track Detectors, etching solutions, and analysis equipment. To describe all the various combinations and processes would be a monumental task. Therefore, this discussion will be limited to the materials and methods used in the standard fission track analysis procedures used by the Environmental Radiation and Toxicology Laboratory of the University of Utah, where the research for this project was performed.

Fission track analysis is performed by the Environmental Radiation and Toxicology Laboratory to detect environmental amounts of ^{235}U or ^{239}Pu in samples of water, reagents, tissue, and human urine and feces. The samples to be tested for ^{235}U or ^{239}Pu content are first chemically processed to separate the uranium and plutonium. The portion of the sample that contains the isotope of interest is then deposited upon a SSNTD. Two different Solid State Nuclear Track Detectors are used to detect fission tracks caused by fission fragments, Lexan and Makrofol polycarbonate. The Lexan and Makrofol are cut in strips 5.5 cm by 1.2 cm and are thoroughly cleaned using ethanol to remove contamination. Then a diamond point scribe is used to scribe three 9 mm circles upon the Lexan. Samples are deposited on the Lexan slide in these circles. After the sample has completely dried, the Lexan slide is covered with a piece of Makrofol. The Lexan - Sample - Makrofol sandwich is then irradiated to a neutron fluence of 1.1×10^{17}. The fissile isotopes in the sample will fission creating two fission fragments. As the Law of the Conservation of Momentum would indicate, one of the fission fragments will pass through the surface of the Lexan and the other fission fragment will pass through the surface of the Makrofol, creating fission tracks in the two SSNTDs. See Figure 1. Therefore, after etching, there should be an identical number of fission tracks in both the Lexan and the Makrofol.

3
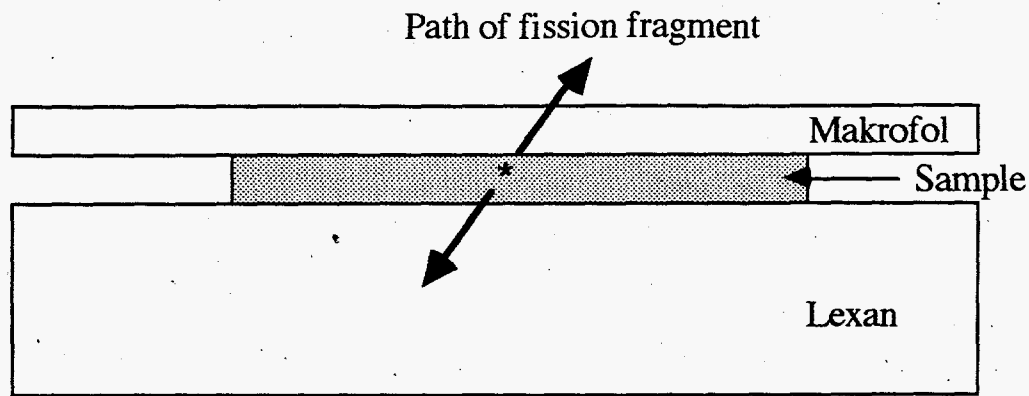
Figure 1. Fission Tracks Occur in Both Lexan and Makrofol

Tracks produced by the passage of fission fragments through solids were first studied in crystals, such as molybdenite, talc, and mica, using transmission electron microscopy [11]. Later, it was discovered that fission tracks were observable in almost all types of dielectric solids. In general, particle tracks can be found and stored in materials that have a resistivity greater than 2×10^5 Ωm [5].

Fission fragments have an enormous potential for causing damage in solids. If a fission fragment were considered an "elementary particle", it would be characterized by having a positive charge of between +30 and +65, an atomic mass of between 65 and 160 amu, and are created in highly excited nuclear and electronic states. Fission fragments have a kinetic energy of about 80 MeV and move at velocities of 1000 km/sec [1]. The energy carried by a fission fragment that is lost to the solid through interactions can be classified as the sum of the energies lost to nuclear processes, radiation processes, and the electronic processes. It is this loss of energies to the surrounding material that creates a fission track.

The loss of energy carried by the fission fragment due to electronic excitation is relatively large in comparison to the other loss mechanisms. These losses are incurred due to the Coulombic attraction of the electrons in the lattice to the highly positive charge of the fission fragment. The passage of the fission fragment through the material's lattice will cause the lattice

electrons to move towards the path of the fission fragment. This will cause bonds in the lattice to be broken and will create a substantial amount of damage to the structure of the lattice. In polymers, such as Lexan and Makrofol, the covalent bonds along the path of the ionizing fission fragment will rupture, causing an area of increased chemical activity and decreased average molecular weight [5].

The rate of the loss of the energy of a fission fragment due to nuclear collisions is negligible until near the end of the fission fragment's path inside the material, where the fragment is moving more slowly [1]. Therefore, the nuclear energy potential carried by the fission fragment does not appreciably contribute to the formation of a fission track. The loss of the energy of a fission fragment due to radiative processes, such as bremsstrahlung and Cherenkov radiation, is nearly negligible.

The theory that models the damage to a crystalline lattice due to the ionizing fission fragment is the lattice instability model. The idea that this model presents is that the damage to a crystal lattice is a result of the mutual repulsion of like ions that become adjacent to each other when lattice electrons are stripped away from the atoms of the crystal lattice due to the positive Coulombic attraction of the passing ionized fission fragment. This theory tends to explain the lack of observable fission tracks in metals, due to the presence of free electrons in metal lattices. Ions created by the passage of a fission fragment through metal would be repaired by free electrons before the mutual repulsion of the positively charged ions would have the chance to do much permanent damage to the structure of the lattice.

The lattice instability model suggests that all materials posses some threshold ionization density below which a particle will not leave a fission track. Hepburn and Windle [5], have listed a few of the more common Solid State Nuclear Track Detectors and have cataloged their sensitivity to ions by the lightest ion that will leave an etchable track. From their data, the lightest detectable ion for polycarbonates, such as Lexan and Makrofol, is Carbon. Hepburn and Windle acknowledge that this data is for polycarbonates in general and that polycarbonates produced by different manufacturing processes will have different sensitivities. Even if some

5

variations in the sensitivity of different polycarbonates exist, this data would indicate that polycarbonate is more than sensitive enough to register tracks from fission fragments.

Another form of fission track detector sensitivity can be observed with fission fragments that are moving at high velocities. Particles will only register in dielectrics over a certain energy range that is dependent upon both the number of nucleons in the particle and the composition of the detector [5]. When a fission fragment impacts the surface of a dielectric solid, it is possible that the fragment may not be moving slowly enough to give the lattice electrons time to react. When this happens, the fragment travels for a distance through the dielectric leaving no visible record of its passage until the fragment begins to slow down enough for the lattice electrons to interact with it. For an example of this, a ^{64}Zn particle with an energy of greater than 100 MeV/nucleon is initially moving too fast to register in polycarbonate and will not begin to leave a track until its energy has been reduced to less than the critical value of 100 MeV/nucleon. In such cases, the etchant must first remove the undamaged surface layers of the dielectric before it can act upon the surface of the track. If the dielectric is under-etched, these fission tracks may be unobservable. This can be overcome by using a dielectric fission track detector that is sufficiently thin for the fission fragment to achieve full penetration. The etchant can then enter the fission track from the back surface of the detector.

Fission Track Etching

Particle tracks were first produced in crystals and observed using an electron microscope, without the benefit of post-exposure etching. It was eventually discovered that the particle tracks could be enhanced if the detector was exposed to a chemical etchant after to its exposure to the particles. These original crystalline particle detectors were etched with extremely reactive chemicals such as HF. Fortunately, the polymer detectors, which proved superior to the crystal detectors at producing tracks from ionized particles, can be etched with chemicals that are substantially safer and easier to use, such as KOH and NaOH. It should be noted that for every different type of SSNTD, there are several ways of etching to develop particle tracks, including both chemical and electro-chemical etching with many different types of etching solutions. This explanation of the mechanisms of etching will be focused upon those particular to the chemical etching of polymer detectors, specifically Lexan and Makrofol polycarbonate.

Etching was found to remarkably enhance the sensitivity of particle detectors to tracks. The primary benefit of track etching is that the action of the chemical etchant upon the detector prevents track fading and annealing. This can be extremely useful because it provides a permanent record of the track in the detector. A second benefit of etching a particle track is in the enhancement of the track. This causes the tracks to be much more easily seen, and facilitates the counting of tracks using optical microscopes in place of an electron microscope. The third benefit to etching tracks is that most tracks that are unobservable before the action of the etchant upon the detector form visible etch channels [11].

The etching solution acts by chemically dissolving the particle track detector. The path of the damage left by the ionizing particle etches preferentially to the undamaged portions of the detector. This preferential chemical dissolving of the particle track creates a channel which is larger than the original damage trail. This etched track is easily observable in a light microscope

7

as a dark conical shaped defect in the detector. This ratio of the length of the conical shaped tracks to their diameter is quite large for most polymer dielectrics.

The preferential action of the etchant in the damaged regions is characterized by the ratio v_T / v_G. v_T is defined as the rate of the chemical etching along the particle path and v_G is the bulk etch rate of the undamaged detector surface [5]. The bulk etch rate and the rate of the etching along the particle path are individualized for different types of detectors and have been experimentally determined for most of the common particle detectors. Polycarbonate etched in 30% KOH (6.5 N) at 70°C has a bulk etch rate value of $v_G = 2.6$ μm/h [12]. If the component of v_T in the direction normal to the surface of the detector is less than v_G, there will be no preferential etching of the track and the track will become obliterated by the action of the etchant. This creates a "critical angle" of incidence for each type of detector, below which no tracks will be apparent after the action of the etchant [8].

If the particle tracks are isotropic and of a finite length in the detector, an etching efficiency can be calculated. The etching efficiency can be defined as the percentage of particle tracks which are visible after exposure of the detector to an etching solution. Polycarbonate has an etching efficiency of 95.7% and the "critical angle" of particle incidence is between 2° 30' and 3.00° [5]. Both of these values show that polycarbonate is a superior track detector compared to glass and quartz detectors which may have efficiencies as low as 42% and "critical angles" as large as 35°.

The temperature and concentration of an etching solution directly influence both the bulk etch rate and the track etch rate. Experimentation has shown that the etching of Lexan for fission tracks will produce very good results when the Lexan is exposed to a 6.5 N KOH solution at 70°C for 11 minutes [4]. The Makrofol used in this project is a thinner detector and is etched in 6.5 N KOH solution at 60°C for 11 minutes to produce the best results. As a note, Hepburn and Windle [5] report that track loss due to annealing occurs in polycarbonate exposed for one hour to 100-110°C etchant and that total track fading occurs in polycarbonate exposed for one hour to etchant at 185-200°C.

8

The response of a plastic track detector to an etching solution can also be influenced by exposure to oxygen, nitrogen, hydrogen peroxide, water, and ultraviolet light before the detector is exposed to the etching solution [7]. Detectors that have been exposed to oxygen before they were exposed to the ionizing particles produce almost twice as many tracks after etching as detectors that are kept in an oxygen-free environment. Detectors that are kept in a nitrogen environment before exposure to ionizing particles produce fewer tracks upon etching than detectors exposed to air. The conclusion that can be drawn from both of these examples is that oxygen is a key component in the formation of particle tracks in detectors.

Hydrogen peroxide has a limited effect upon the sensitivity of plastic detectors to ionizing particles, but it does have a rather considerable effect upon the etch rate of the detector [7]. Detectors that are exposed to H_2O_2 before exposure to ionizing particles produce a slightly larger number of fission tracks than detectors that have not been exposed to the H_2O_2. On the other hand, detectors that have been treated with H_2O_2 have a much higher etch rate than non-exposed detectors. Khan suggests that this is due to a "softening" effect of the H_2O_2 on the detector that decreases the detector's resistance to the chemical effects of the etchant.

The exposure of a detector to water has much the same effect upon the sensitivity of the detector to particle tracks and the etch rate of the detector as does H_2O_2. Khan [7] also suggests that this may be caused by the water lowering the resistance of the detector to the chemical action of the etchant.

Detectors that have been exposed to ultraviolet light both before exposure to the ionizing particles and before etching have shown greatly enhanced etching rates. The exposure of polycarbonate to UV light in the presence of oxygen has resulted in an increase in the etch rate as high as a factor of 10^4 [9]. The unfortunate side-effect to this is that if the exposure of the detector to the UV light is uneven, the etching of the particle tracks is also going to be uneven. Therefore it is with caution that one should use UV light to increase the etching rate.

The etch rate of a detector is also subject to variation due to the production of an "etch product layer". This layer is composed of the dissolved surface of the detector and forms on the

surface during the etching process [7]. This layer can form a shield on the surface of the detector, the effectiveness of which increases with thickness. This "etch product layer" can be minimized by stirring the etching solution and by minimizing the etching time. For this project, the Lexan detectors were stirred in the etching solution, producing the same effect.

Another factor that influences the etching of the detectors is mentioned by Khan [7]. Oils that are deposited on the surface of a detector due to handling are hard to remove without scratching the detector's surface. These oils collect etchant and etch products during etching which interferes with the uniform etching of the detector surface. In addition to this, these marks can also interfere with the viewing of the tracks. Therefore, the detectors should be carefully handled using gloves to avoid fingerprints. The detector should also be thoroughly rinsed in water after etching to remove all etchant and etchant products. The rinsed track detectors should be allowed to air dry rather than dried with a tissue. Tissue scratches the surface of the detector and affects the surface quality.

After the detectors are etched and rinsed, they can be viewed to count the particle tracks. In this project, the Lexan fission track detectors are mounted upon glass microscope slides for ease of viewing. After the Makrofol is etched, rinsed and thoroughly dried, it is counted in the Spark Gap Chamber. It can be noted at this time that after irradiation and etching, the Lexan detectors will be an opaque amber color in contrast to the clear translucent nature of unirradiated, unetched Lexan. The exposure of the Lexan to the radiation in the reactor causes the diffusion of O_2 into the polymer matrix, causing it to slowly turn an amber color. The particle flux in the reactor also creates a great deal of surface pitting in the Lexan that is enhanced by etching, causing the Lexan to become opaque after etching. These surface pits in the Lexan are clearly visible in the microscope. Etched Lexan can be seen in Figure 2. Fission tracks appear as dark shapes against the mottled background of the surface pitting of the detector.
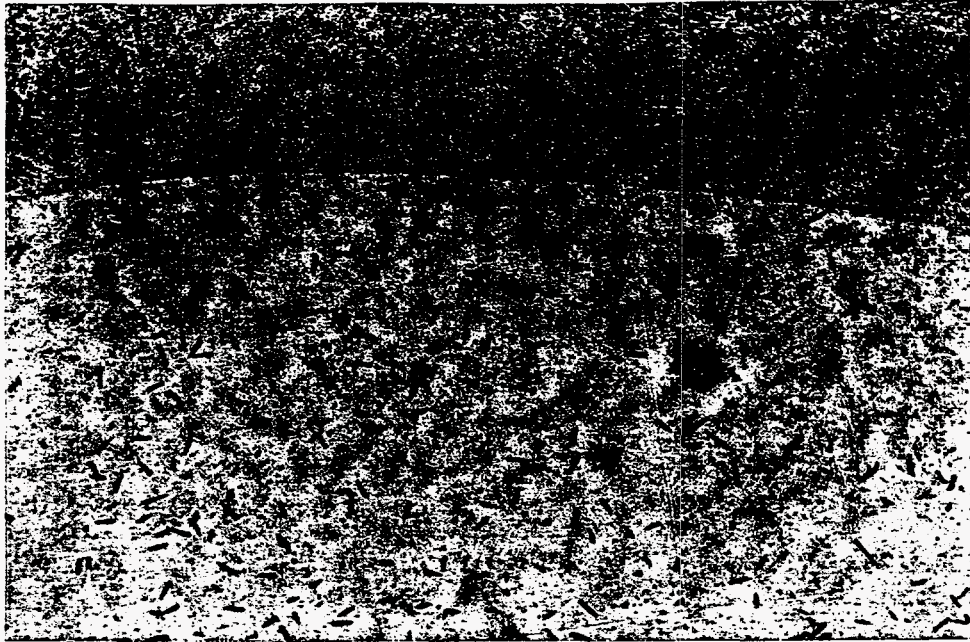
Scale: 1 cm = 56 μm
Microscope Magnification: x 100
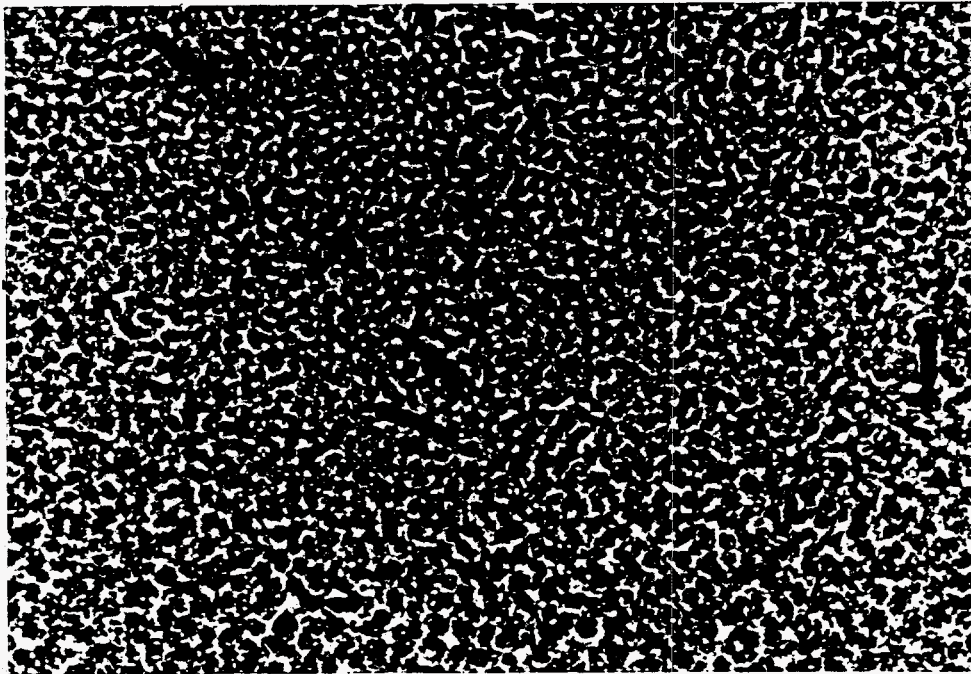
Scale: 1 cm = 13 μm
Microscope magnification :   x 400

Figure 2. Etched Fission Tracks in Lexan

11

Spark Chamber

Particle tracks can be counted in several different ways, including the original method of counting unetched tracks using an electron microscope. Etched fission tracks in polycarbonate detectors are large enough to be identified using an optical microscope. However, the manual counting of fission tracks is labor intensive and subject to human error. Therefore, much research has been done to develop a quick, reliable way to count fission tracks. One method that has been developed uses an automated image analyzer, such as the Quantimet 720 Image Analyzer [10]. Another method uses the Spark Chamber.

The spark counting technique uses an etched dielectric detector foil as the insulator between two electrodes. Dielectric breakdown will occur across the etched fission tracks resulting in a spark that can be counted using a pulse analyzer. In this manner, the fission tracks in a dielectric detector can be counted in a matter of a few seconds, versus the hours of time that it would require to count the tracks visually using a microscope. A simplified circuit diagram for the spark chamber used in this project is given on page 3 of the appendix.

The spark chamber used in this project is constructed of a combination of Plexiglas and brass. The construction of the spark chamber and its electronic components is detailed in the appendix. A cut-away side view of the spark chamber is given in figure 3. This spark chamber was cross calibrated with another spark chamber design by Ingrao, et al. [6].

The electrode used in the spark chamber for this project is brass with a highly polished, flat surface. This electrode is connected to the negative terminal of a high voltage power supply. The outer brass ring of the spark chamber is connected to the positive terminal of the high voltage power supply and supplied with a negative voltage. Dry, etched Makrofol is placed upon the polished electrode and covered with a piece of aluminized Mylar. The Mylar is placed with the aluminized side in simultaneous contact with the outer brass ring of the spark chamber and the Makrofol, and serves as the positive electrode.
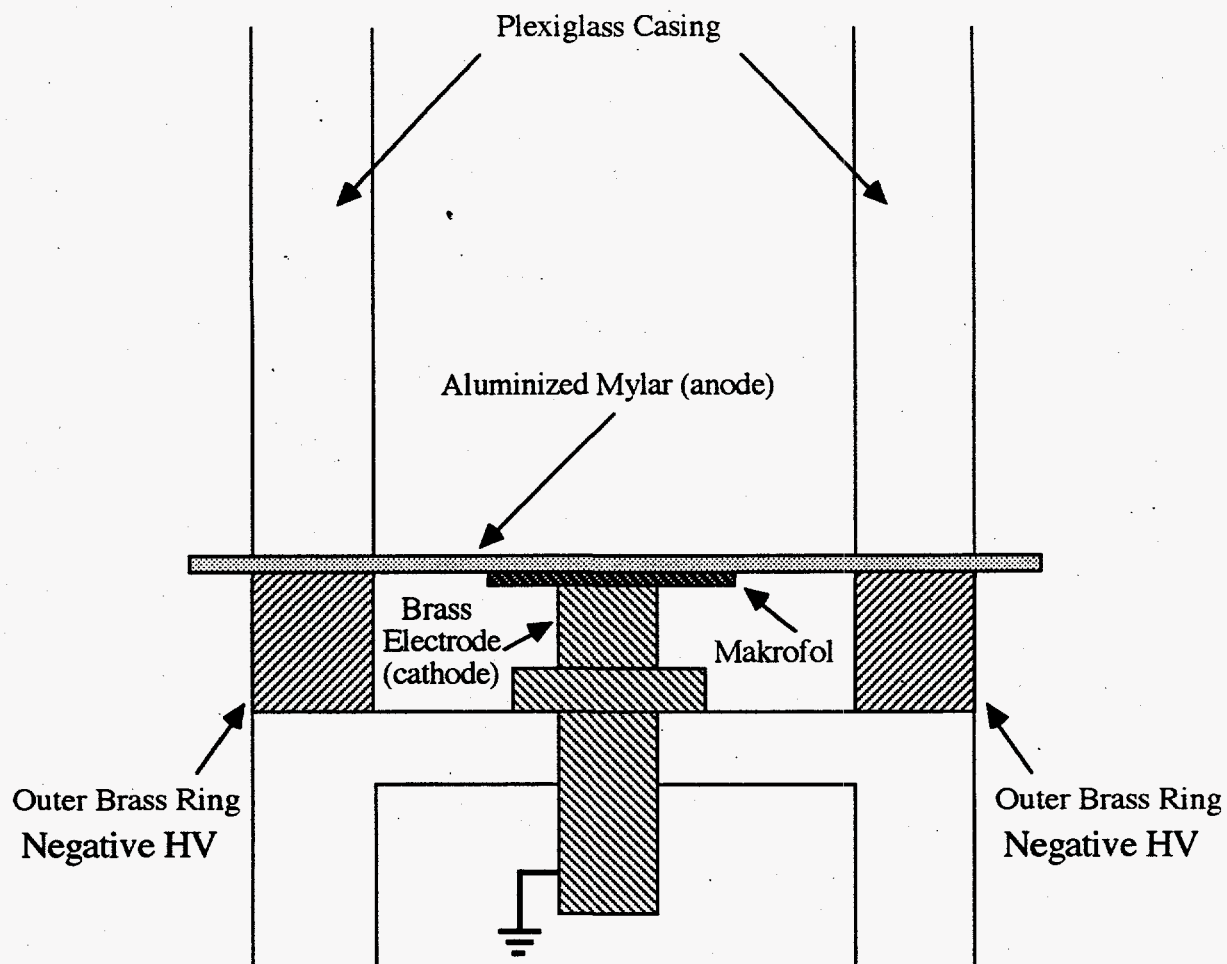
Figure 3. Spark Chamber Design

Makrofol polycarbonate with a thickness of 10 μm is used as the dielectric detector for samples that are to be counted in the spark chamber. Makrofol was chosen as the detector of choice for several reasons. First, Makrofol is produced as a thin foil. This is desirable because the breakdown voltage for Makrofol is low enough that it can be used in a spark chamber without the dangers inherent to a high voltage system. In addition to the thinness of the Makrofol foil, the polycarbonate construction of the foil is a desirable quality. These two qualities of the Makrofol foil allow for the fission fragments to cross the entire thickness of the foil [14]. The

13

subsequent etching of the foil produces fission tracks that completely perforate the Makrofol and allow for dielectric breakdown of the foil along the path of the fission track.

The Mylar used in the spark chamber for this project is aluminized on one side. Since a new piece of Mylar is used for each operation of the spark chamber, it is important for comparison reasons that the resistivity of the Mylar should be constant for all samples counted in a spark chamber. The Mylar used in the spark chamber for this project has a resistivity of approximately 2.5 Ω/square.

Before the Makrofol can be counted in the spark chamber, it is important that the etched fission tracks completely penetrate the foil. Tracks that do not penetrate require breakdown voltages that may be higher than the voltage used to count the other fission tracks. These non-penetrating tracks may therefore not be counted during the sparking process and may result in a loss of counting efficiency. These tracks can be enlarged so that they completely perforate the foil by applying a higher pre-counting voltage, commonly called a "pre-sparking" voltage [15]. All samples counted in the spark chamber for this project undergo a pre-sparking to increase the efficiency of the counting process. This pre-sparking voltage is set at 1200 V for this project and the normal spark counting voltage is 500 V [4].

After the etched Makrofol is thoroughly rinsed and dried, it can be pre-sparked and then spark counted in the spark chamber. The sample area of interest on the Makrofol is identified and centered on the spark chamber electrode. The Makrofol can be placed with either side on the polished brass electrode since the breakdown will occur across incompletely penetrating tracks regardless of whether the blockage is at the end of the path of the spark or at the beginning of the spark's path. Aluminized Mylar is placed such that the aluminized side completely covers the portion of the Makrofol that is in contact with the brass electrode and such that the Mylar is also in contact with the outer brass ring of the spark chamber. It is important that the entire portion of the Makrofol on the electrode is covered by the aluminized side of the Mylar because the Mylar acts as the positive electrode in the circuit. The Makrofol must be in good contact with the polished brass electrode and with the Mylar for both pre-sparking and spark counting. This is

14

accomplished by using an insulated plunger to press the Mylar and Makrofol firmly together and onto the brass electrode.

The polished brass electrode and the outer brass ring of the spark chamber are connected to the high voltage power supply in the manner shown in figure 3 and supplied with a negative voltage. When the negative high voltage is supplied to the spark chamber, a spark will travel through one of the etched fission tracks in the Makrofol with enough energy to evaporate the aluminum on the Mylar in a hole of a size larger than the fission track. The diameter of these evaporated holes in the aluminized Mylar is between 50 μm to 200 μm for an applied spark counting voltage of 500 V. These evaporated holes are much larger for an applied pre-sparking voltage of 1200 V [4].

The spark is rapidly stopped by the discharge of the capacitor in the spark chamber circuit. After the spark has been quenched by the capacitor, it cannot travel through the same hole in the Mylar because the path to the edge of the hole in the aluminum is too long [3]. Therefore, the spark will travel through another hole in the Makrofol to complete the circuit. The discharge of the capacitor will stop the spark through this hole as well. In this manner, the spark will continue jumping from one hole in the Makrofol to another until it has jumped through all the holes. The discharge of the system will stop and the voltage supply can be turned off. The counting process will be completed in approximately 20 seconds after the voltage is supplied to the spark chamber, in comparison to the 30 minutes or longer that is required to count the number of fission tracks in the Lexan from the same sample.

The sparking process develops the Mylar into a replica of the holes in the Makrofol, which can be seen with the human eye. It should be noted that a new piece of aluminized Mylar must be used for each pre-sparking and for each spark counting of a sample because of the holes developed in the aluminum layer during the sparking process. Ideally, the number of holes in the Mylar should be exactly equal to the number of spark pulses that were electronically counted and equal to the number of fission track holes in the Makrofol. However, it is possible for a spark to travel through a fission track hole multiple times. This will occur if the evaporated hole in the

15

aluminum layer on the Mylar is not large enough to prevent a spark from traveling through the fission track hole a second time. This phenomenon is directly related to the voltage applied to the spark chamber. At high voltages, such as the pre-sparking voltage, this occurs quite frequently. The spark counting voltage must be chosen to minimize the effect of the "double sparking" of the fission track holes while also maximizing the number of fission track holes in the Makrofol which are sparked.

Another factor that influences the results from the spark chamber occurs in samples that have a high density of fission tracks. At high track densities, fission tracks may overlap. These overlapping tracks can be pre-sparked into one large hole in the Makrofol. This large hole will be sparked once and will count as one fission track hole when there may have been many tracks at this location. A high density of fission tracks may also spark in such a manner as to produce electrically insulated islands in the aluminized Mylar [13]. Fission track holes in the Makrofol that lie underneath this insulated island will not be spark counted. This will therefore lower the efficiency of the spark chamber.

Chatham, et al. give a list of the factors to which the spark chamber is extremely sensitive [2]. Included in this list are factors involving defects in the polycarbonate detector, non-uniformity in the deposition of the aluminum on the Mylar, and non-uniform fission track density. These factors should be eliminated or minimized as is possible.

Another factor that is of concern when using a spark counting system concerns the waiting time between when the detector is exposed to the fission fragments and when it is etched. The intensity of the damage to the polycarbonate detectors is increased with time. This is important for the spark chamber because the increased damage may facilitate the growth of several overlapping fission tracks into one larger hole. Khan [7] suggests that the waiting time between exposure and etching should be standardized. This suggestion was used in the development of the procedure for etching Makrofol given in this paper.

16

The spark chamber is sensitive to a number of factors that will affect its counting efficiency. In spite of this, the spark chamber still a valuable tool for counting fission tracks because it allows for a rapid reading of a fission track detector.

Discussion of Procedures

Although there has been much research published on the various methods and materials of fission track analysis using Solid State Nuclear Track Detectors, there has been little published on the procedures used to etch Lexan and Makrofol to develop fission tracks and on the procedures used to operate a Spark Chamber. These procedures are presented here in a format that allows the reader to consistently develop good fission tracks in Lexan and Makrofol and allows the reader to understand the operation of the Spark Chamber used by the researcher to count fission tracks in Makrofol.

Lexan Etching Procedure

Purpose and Scope:

This procedure describes the etching process for developing fission tracks in irradiated Lexan slides.

Equipment and Materials:

(1) Irradiated Lexan slides

(2) Temperature Controlled Water Bath with Teflon covered aluminum top

(3) Three 2 liter Pyrex beakers

(4) Stir drill on Teflon covered stand

(5) Stir drill bit

(6) 24 pieces of clean Lexan, 5.5 cm x 1.2 cm x 0.25 mm

(7) Two white plastic report cover binders

(8) Metal file

(9) Duro Quick Gel Super Glue

(10) Digital Timer

(11) Latex gloves

(12) 150 ml ethyl alcohol

(13) Glass Microscope slides

(14) Scotch Tape

(15) Paper towels

(16) Deionized water

Procedure:

The process of etching irradiated Lexan slides to develop the fission tracks requires many preparatory steps that need to be taken before the Lexan can be etched in the KOH solution. The first of these preparatory steps involves the making of a holder for the Lexan slides so that they can be uniformly etched in the KOH. The Lexan slide holder is designed such that the KOH solution is circulated around the Lexan slides in a manner that allows for the uniform exposure of all slides to the KOH solution.

The holder for the Lexan consists of pieces of report cover binder glued onto a Teflon stir drill bit. Two plastic report cover binders are each cut into three sections of equal length. Any rough sections on the pieces of binder should be filed off to protect the Lexan from scratches during etching. Using a metal file, the Teflon stir drill bit is scraped to roughen the surface. The stir drill bit and the pieces of report cover binder are then prepared by thoroughly cleaning in ethanol. These steps help insure that the pieces of report cover binder adhere to the surface of the Teflon stir drill bit. The six pieces of report cover binder can then be glued onto the sides of the stir drill bit as near to the bottom end as possible, using the Duro Quick Gel Super Glue. Figure 4 displays the top view of the stir drill bit with the report cover binders attached. Duro Quick Gel Super Glue is the best adhesive for this assembly due to its resistance to the KOH solution and its ability to adhere to the Teflon coating on the stir drill bit. The glue must be allowed to dry overnight before use. Then the stir drill bit can be firmly clamped into the stir drill.

Fresh KOH must be used for each set of 50 samples to insure uniform etching of the Lexan. Etching requires 6.5 N KOH, which is made by adding 832g of KOH pellets to 2 liters of deionized water. When the KOH pellets are mixed with the water, an exothermic reaction takes

20

place that can release caustic KOH vapor into the air. Therefore, the mixing of the KOH pellets and the water should be done in an ice bath in a fume hood and the KOH pellets should be slowly added to the deionized water to minimize the formation of KOH vapors.

Report cover binders

Teflon stir rod
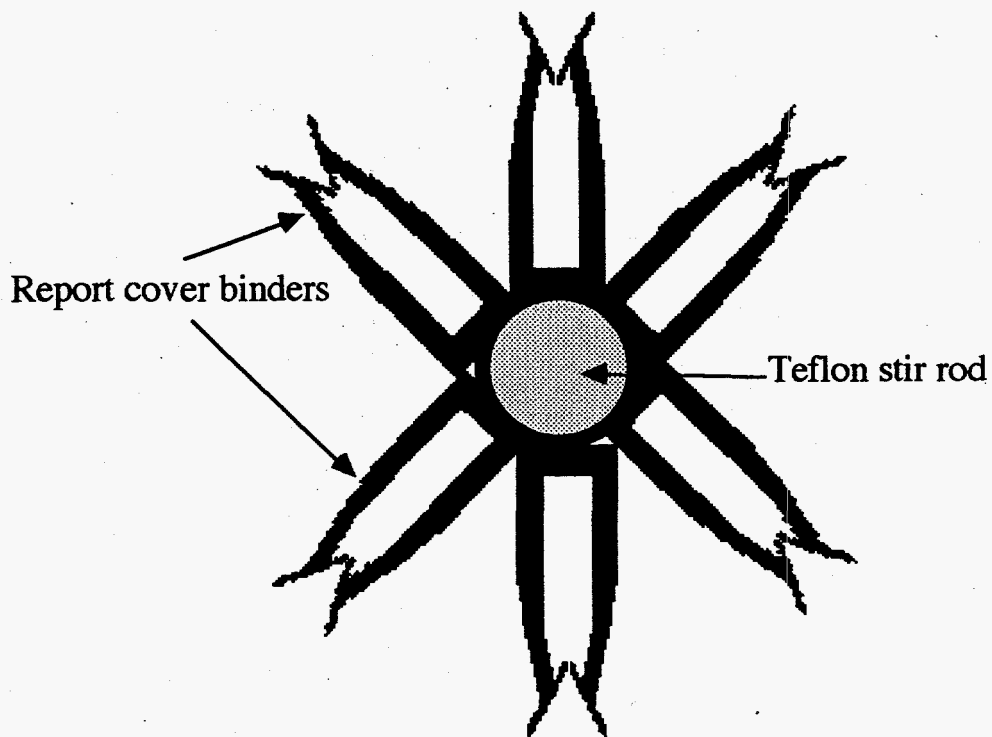
Figure 4. Stir Drill Holder

The temperature controlled water bath is cleaned of mineral deposits and placed in a fume hood. A Teflon covered aluminum top is placed on the water bath such that the cut-out hole for the beaker of etching solution is easily accessible. The three 2 liter beakers must be thoroughly cleaned and rinsed in deionized water to prevent contamination of the Lexan slides with mineral deposits and other foreign substances. One of the 2 liter beakers is then filled with 1.4 liters of the fresh 6.5N KOH solution and placed in the water bath. The water bath can then be filled with water until the water level is slightly over the level of the KOH in the beaker, but not to more

than to the 1.6 liter mark on the beaker. Keeping the level of the water in the water bath above the level of the KOH insures that the entire beaker of KOH solution is completely and uniformly heated to 70°C and helps avoid large temperature gradients in the KOH. However, the water level in the water bath should not be high enough to damage the components of the water bath heater. The water bath can then be turned on and set to warm the water to slightly above 70°C.

The KOH solution is heated to a temperature of 70°C, as measured by a mercury thermometer placed in the beaker of solution. The temperature of the KOH solution must be maintained at 70°C throughout the etching procedure, and the water level of the water bath must remain slightly above the level of KOH in the beaker. Another consideration is the evaporation of water in the KOH solution during the etching process. Usually this is not much of a factor, however it can become a concern if there are many Lexan slides to be etched and the etching process takes more than 3 hours to complete. This problem can be overcome by adding deionized water to the KOH solution to return the volume of the solution to 1.4 liters. After adding the deionized additional water to the KOH solution, the solution must be thoroughly mixed and allowed to reheat to a temperature of 70°C.

After the KOH solution has been placed into the water bath to heat, the rest of the etching apparatus can be assembled. The stir drill is firmly clamped onto the Teflon covered stand and the stand is placed next to the water bath. The stir drill bit is then firmly clamped into the stir drill and the stand is positioned such that the drill bit is in the center of the beaker when it is lowered into the KOH. The other 2 liter beakers are then filled with 1.8 liters of deionized water for rinsing the etched Lexan, and positioned near the Teflon covered stand such that the drill bit can be easily lowered into one of the beakers of rinse water. The second beaker of deionized rinse water is used for rinsing the Lexan slide holders between uses.

The 24 pieces of clean, unirradiated Lexan are rinsed in ethanol and allowed to air dry. These pieces of Lexan are used to help clamp the irradiated Lexan firmly into the holders on the stir drill bit and must be clean to avoid leaving foreign deposits on the irradiated Lexan during etching. The irradiated Lexan also must not be touched with bare hands at any time. This

22

protects the Lexan from scratches and foreign deposits and prevents radioactive contamination and KOH from coming into contact with the hands. Therefore, it is important for gloves to be worn at all times during the rest of the etching procedure.

The Lexan is etched in batches of six each, one piece of irradiated Lexan per holder on the stir drill bit. One of the half pieces of Lexan should be placed on either side of the irradiated Lexan along the longest edge to form a sandwich, such that the half-pieces of clean Lexan covers at least 5 mm of the Lexan from the edge, but not covering any portion of the sample drops on the irradiated Lexan. Figure 5 shows the placement of the Lexan spacers on the irradiated Lexan. The Lexan sandwich is then slid into one of the holders on the drill bit, and positioned so that the bottom of the sandwich is approximately 3.5 cm above the bottom of the bit. Using the same technique, the other five holders in the stir drill bit are filled.

Lexan Spacers

R18-26 ◄———————— Slide code marked in ink

——— Sample Drops

Lexan or Makrofol
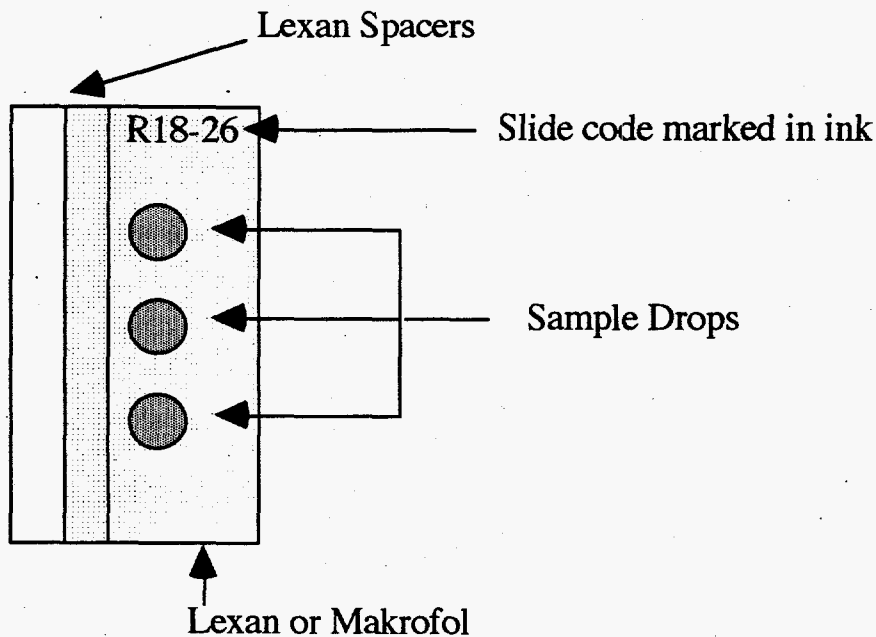
Figure 5. Placement of Lexan Spacers

The stir drill equipped with the loaded bit is lowered into the 70°C KOH until the bottom of the bit is just above the bottom of the beaker and the Lexan slides are completely submerged in the KOH. The stir drill is then set to stir on a low speed and the Lexan is left in the 70°C KOH solution for 11 minutes. It is important to note that the super glue that was used to attach the report cover binders onto the stir drill bit will slowly dissolve in the KOH and turn the solution an orange color. This does not significantly affect the etching of the Lexan, but the super glue will be extremely brittle after the etching of approximately 48 Lexan slides, and it will become easy to break the report cover binders off the Teflon stir drill bit. Therefore, if large batches of Lexan slides are to be etched, more than one stir drill bit may be required to be prepared before the beginning of the etching process.

After 11 minutes has elapsed, the stir drill is turned off and quickly moved so that the bit can be lowered into the center of one of the 2 liter beakers of deionized rinse water. The Lexan is stirred on a low speed in the rinse water for approximately 5 minutes. Then the drill is turned off, raised on the stand, and moved such that the stir drill bit is removed from the beaker of rinse water. The irradiated Lexan and the half-pieces of Lexan are carefully removed from the bit and rinsed by hand under a running stream of deionized water for about one minute.

The irradiated Lexan should then be briefly dipped into a 150 ml beaker of ethyl alcohol such that the alcohol covers all the sample areas but does not touch the portion of the Lexan that has the slide identification code marked on it. This prevents the alcohol from dissolving the ink that was used to mark the code on the Lexan slides. After the Lexan is removed from the alcohol it is placed on a clean, dry paper towel and allowed to air dry.

For the final step in the etching process, the dry, etched Lexan slides need to be placed onto clean glass slides to facilitate the counting of the fission tracks through an optical microscope. The Lexan slides are carefully taped into place on a clean glass slide such that the tape is not placed over any portion of the sample drops. The glass slides are to be handled by the edges only and are placed in a microscope slide box for storage.

Procedure for Etching and Spark Counting of Makrofol

Purpose and Scope:

This procedure describes the spark counting process in two parts. Part one describes the etching to develop fission tracks in irradiated Makrofol. Part two describes the operation of the spark chamber to count the fission tracks in the etched Makrofol.

Equipment and Materials:

(1) Irradiated Makrofol

(2) Temperature Controlled Water Bath with Teflon covered aluminum top

(3) Three 2 liter Pyrex beakers

(4) Stir drill on Teflon covered stand

(5) Stir drill bit

(6) 24 pieces of clean Lexan, 5.5 cm x 1.2 cm x 0.25 mm

(7) Two white plastic report cover binders

(8) Metal File

(9) Duro Quick Gel Super Glue

(10) Digital Timer

(11) Latex gloves

(12) 150 ml ethyl alcohol

(13) Paper towels

(14) Deionized water

(15) Spark Chamber and 8 mm diameter brass electrode

(16) Brasso Polishing Compound

(17) Buehler Microcloth

(18) EG&G Ortec NIM Bin and power supply

(19) EG&G Ortec 556 High Voltage Power Supply

(20) EG&G Ortec 770 Counter

(21) EG&G Ortec 771 Timer-Counter

(22) EG&G Ortec 590A Amplifier

(23) Voltage Controller

(24) Spark Chamber Interface Circuit

(25) Aluminized Mylar

(26) Razor blade for cutting Mylar

(27) Small cutting board

(28) Sheet protectors

(29) Black "Sharpie" marking pen

(30) Black ball-point pen

Procedure:

Part 1: Procedure for etching irradiated Makrofol.

Before counting the fission tracks on the irradiated Makrofol using the Spark Chamber, it is necessary to first etch it in a 6.5 N. KOH solution. The procedure for etching the Makrofol is essentially the same as the procedure for etching irradiated Lexan, with a few changes.

An important item that should be noted is that the Makrofol need not be etched on the same day as the irradiated Lexan slides. However, the Makrofol must be etched within 5 days of their

irradiation. This restriction is necessary to minimize the annealing of the fission tracks in the Makrofol.

The process of etching irradiated Makrofol to develop the fission tracks requires many preparatory steps that need to be taken before the Makrofol can be etched in the KOH solution. The first of these preparatory steps involves the making of a holder for the pieces of Makrofol so that they can be uniformly etched in the KOH. The Makrofol holder is designed such that the KOH solution is circulated around the Makrofol in a manner that allows for the uniform exposure of all pieces to the KOH solution.

The holder for the Makrofol consists of pieces of report cover binder glued onto a Teflon stir drill bit. Two report cover binders are each cut into three sections of equal length. Any rough sections on the pieces of binder should be filed off to protect the Makrofol from scratches, rips, and tears during etching. Using a metal file, the Teflon stir drill bit is scraped to roughen the surface. The stir drill bit and the pieces of report cover binder are then prepared by thoroughly cleaning in ethanol. These steps help insure that the pieces of report cover binder adhere to the surface of the Teflon stir drill bit. The 6 pieces of report cover binder can then be glued onto the sides of the stir drill bit as near to the bottom end as possible, using the Duro Quick Gel Super Glue. The glue must be allowed to dry overnight before use. Then the stir drill bit can be firmly clamped into the stir drill.

Fresh KOH must be used for each set of 50 samples to insure uniform etching of the Makrofol. Etching requires 6.5 N. KOH, which is made by adding 832g of KOH pellets to 2 liters of deionized water. When the KOH pellets are mixed with the water, an exothermic reaction takes place that can release caustic KOH vapor into the air. Therefore, the mixing of the KOH pellets and the water should be done in an ice bath in a fume hood and the KOH pellets should be slowly added to the deionized water to minimize the formation of KOH vapors.

The temperature controlled water bath is cleaned of mineral deposits and placed in a fume hood. A Teflon covered aluminum top is placed on the water bath such that the cut-out hole for the beaker of etching solution is easily accessible. The three 2 liter beakers must be thoroughly

27

cleaned and rinsed in deionized water to prevent contamination of the Makrofol with mineral deposits and other foreign substances. One of the 2 liter beakers is then filled with 1.4 liters of the fresh 6.5N KOH solution and placed in the water bath. The water bath can then be filled with water until the water level is slightly over the level of the KOH in the beaker, but not to more than to the 1.6 liter mark on the beaker. Keeping the level of the water in the water bath above the level of the KOH insures that the entire beaker of KOH solution is completely and uniformly heated to 60°C and helps avoid large temperature gradients in the KOH. However, the water level in the water bath should not be high enough to damage the components of the water bath heater. The water bath can then be turned on and set to warm the water to slightly above 60°C.

The KOH solution is heated to a temperature of 60°C, as measured by a mercury thermometer placed in the beaker of solution. The temperature of the KOH solution must be maintained at 60°C throughout the etching procedure, and the water level of the water bath must remain slightly above the level of KOH in the beaker. It is more important for the temperature of the KOH to be exact for the etching of Makrofol than it is for the etching of Lexan because the Makrofol is a much thinner plastic and is extremely sensitive to the variation of temperature during etching. Another consideration is the evaporation of water in the KOH solution during the etching process. Usually this is not much of a factor, however it can become a concern if there are many Lexan slides to be etched and the etching process takes more than 3 hours to complete. This problem can be overcome by adding deionized water to the KOH solution to return the volume of the solution to 1.4 liters. After adding the deionized additional water to the KOH solution, the solution must be thoroughly mixed and allowed to reheat to a temperature of 60°C.

After the KOH solution has been placed into the water bath to heat, the rest of the etching apparatus can be assembled. The stir drill is firmly clamped onto the Teflon covered stand and the stand is placed next to the water bath. The stir drill bit is then firmly clamped into the stir drill and the stand is positioned such that the drill bit is in the center of the beaker when it is lowered into the KOH. The other 2 liter beakers are then filled with 1.8 liters of deionized water

for rinsing the etched Makrofol, and positioned near the Teflon covered stand such that the drill bit can be easily lowered into one of the beakers of rinse water. The second beaker is used as a second rinse for rinsing the etched Makrofol.

The 24 pieces of clean, unirradiated Lexan are rinsed in ethanol and allowed to air dry. These pieces of Lexan are used to help clamp the irradiated Makrofol firmly into the holders on the stir drill bit and must be clean to avoid leaving foreign deposits on the irradiated Makrofol during etching. The irradiated Makrofol also must not be touched with bare hands at any time. This protects the Makrofol from scratches, rips, tears and foreign deposits and prevents radioactive contamination and KOH from coming into contact with the hands. Therefore, it is important for gloves to be worn at all times during the rest of the etching procedure.

The Makrofol is etched in batches of 6 each, one piece of irradiated Makrofol per holder on the stir drill bit. The irradiated Makrofol is stored in a plastic sheet protector after it has been unpacked from the irradiation container. The pieces of Makrofol to be etched should first be examined to find the areas corresponding to the locations of the sample drops. One of the half pieces of Makrofol should be placed on either side of the irradiated Makrofol along the longest edge to form a sandwich, such that the half-pieces of clean Makrofol covers at least 5 mm of the Makrofol from the edge, but not covering any portion of the sample drops on the irradiated Makrofol. The Lexan-Makrofol sandwich is then slid into one of the holders on the drill bit, and positioned so that the bottom of the sandwich is approximately 3.5 cm above the bottom of the bit. Using the same technique, the other 5 holders in the stir drill bit are filled.

The stir drill equipped with the loaded bit is lowered into the 60°C KOH until the bottom of the bit is just above the bottom of the beaker and the Lexan-Makrofol sandwiches are completely submerged in the KOH. The stir drill is then set to stir on a low speed and the Makrofol is left in the 60°C KOH solution for 11 minutes. It is important to note that the super glue that was used to attach the report cover binders onto the stir drill bit will slowly dissolve in the KOH and turn the solution an orange color. This does not significantly affect the etching of the Makrofol, but the super glue will be extremely brittle after the etching of approximately 50 pieces of Makrofol,

29

and it will become easy to break the report cover binders off the Teflon stir drill bit. Therefore, if large batches of irradiated Makrofol are to be etched, more than one stir drill bit may be required to be prepared before the beginning of the etching process.

After 11 minutes has elapsed, the stir drill is turned off and quickly moved so that the bit can be lowered into the center of one of the 2 liter beakers of deionized rinse water. The Makrofol is stirred on a low speed in the rinse water for approximately 5 minutes. Then the Makrofol is rinsed for 10 minutes in the second beaker of fresh deionized water. Following this, the drill is turned off, raised on the stand and moved such that the stir drill bit is removed from the second beaker of rinse water. The irradiated Makrofol and the half-pieces of Lexan are removed from the bit and rinsed by hand in a lab sink under a slow running stream of deionized water for about one minute.

The irradiated Makrofol should then be briefly dipped into a 150 ml beaker of ethyl alcohol such that the alcohol covers all the sample areas but does not touch the portion of the Makrofol that has the slide identification code marked on it. This prevents the alcohol from dissolving the ink that was used to mark the code on the Makrofol pieces. The Makrofol is removed from the alcohol, placed on a clean, dry paper towel and allowed to air dry. After the Makrofol is dry, it is stored in a clean sheet protector until it is counted in the Spark Chamber.

Part 2: Procedure for operating the Spark Chamber.

The second part of this procedure is to count the fission tracks in the etched Makrofol using the Spark Chamber. There are some preparatory steps to be taken before the Makrofol can be spark counted. The first of these steps involves the preparation of the Spark Chamber and its electrode.

An electrode that is slightly larger in diameter than the sample drop on the Makrofol is chosen for use in the Spark Chamber. The electrode used for counting the Makrofol in this

project is an 8 mm diameter brass electrode. To insure good contact between the Makrofol and the electrode it is important that the electrode be flat and highly polished. The brass electrodes can be made flat by using a diamond wafering blade to cut a flat surface on the top of the electrode. The surface on the electrode is then carefully polished using Brasso and Buehler Microcloth until it is smooth and mirror-like. The electrode must then be handled with care to avoid damaging this surface.

The now flat electrode should be carefully polished again just before using it in the Spark Chamber to remove any oxidation on its surface. The electrode is lightly polished using Brasso and Buehler Microcloth and is cleaned using ethanol and a clean, particle-free cloth. The Spark Chamber itself should be polished with Brasso and a soft cloth to remove any oxidation and to insure good contact between the Mylar and the outer brass ring that serves as the other electrode in the circuit. After polishing, the electrode and the brass ring on the Spark Chamber should not be touched with bare hands, to minimize contamination and further oxidation.

A large supply of aluminized Mylar is cut into strips approximately 6.3 cm x 2 cm before beginning to spark count the Makrofol. The Mylar should not be touched without using forceps or gloves. This is important because dust and oils can change the electrical resistance of the Mylar. The Mylar that is used in the Spark Chamber is cut from the same roll to ensure that all Mylar used in the Spark Chamber has approximately the same electrical resistance. Mylar is cut from the roll using a clean razor blade and a small polyethylene cutting board that has been cleaned with ethanol, and stored flat in clean plastic bags until usage. During the spark counting process it is not necessary to wear latex gloves because the Makrofol and Mylar can be manipulated with forceps during the entire process.

Once the etched Makrofol is completely dry, it is then possible to count the fission tracks in the Makrofol using the Spark Chamber. The Spark Chamber is prepared by placing the polished electrode into its recess in the Spark Chamber. The Spark Chamber is then connected to the Spark Chamber Interface Circuit with the brass electrode functioning as the anode and the outer brass ring of the Spark Chamber as the cathode in a negative voltage system. The power supply

to the EG&G Ortec Nuclear Instrument Module "NIM" Bin, which supplies the power to the 770 Counter, the 771 Timer-Counter, and the 590A Amplifier, is turned on and allowed to warm up for 5 minutes before use.

Before the 556 High Voltage Supply is turned on, the switch on the Voltage Controller should be in the "Discharge" position and the 556 High Voltage Supply output should be set to zero volts to prevent an accidental discharge of voltage. Then the High Voltage Power Supply is turned on and allowed to warm up for 5 minutes before usage. The output meter on the High Voltage Power Supply is also checked to insure that it reads zero volts.

The next step in this procedure is to set the pre-sparking voltage on the Voltage Controller. It is important to know and understand the functions for the various dials and switches on the Voltage Controller to understand how to regulate the voltage output to the Spark Chamber. [See Appendix, "Development of a Spark Gap System to Quantitate Fission Fragment Tracks in Polycarbonate Foil" by E. Martin Isenberg.]:

(1) "HIGH - LOW" Switch:

      "HIGH" -- to set pre-sparking voltage, maximum voltage approximately 1480 V.

      "LOW" -- to set spark counting voltage, maximum voltage approximately 680 V.

(2) "COARSE" Dial: Makes coarse adjustments to the voltage applied to the Spark Chamber.

(3) "FINE" Dial: Males fine adjustments to the voltage applied to the Spark Chamber.

(4) "RATE" Dial: adjusts voltage buildup time. Quicker buildup times are obtained by turning the "Rate" dial counter-clockwise.

(5) "NEGATIVE" - "POSITIVE" Switch: Along with the corresponding changes in polarity switches on the 556 High Voltage Power Supply and on the 590A Amplifier, the settings on this switch have the following impact on the Spark Chamber:

"NEGATIVE" --   in this configuration, the current flows from the brass electrode through the Makrofol and to the Mylar, which serves as a reference to ground. This is the standard configuration for the Spark Chamber.

"POSITIVE" --   in this configuration, the settings on the 556 High Voltage Power Supply and the 590A Amplifier are switched so that the Spark Chamber operates with the current flowing from the Mylar through the Makrofol and to the brass electrode, which serves as a reference to ground.

(6) "START - HOLD - DISCHARGE" Switch:

"START" -- applies voltage to Spark Chamber.

"HOLD" --   holds the voltage applied to the Spark Chamber to the currently displayed level.

"DISCHARGE" --   reduces voltage applied to the Spark Chamber to zero.

To set the pre-sparking voltage, the "HIGH - LOW" switch should be set to "HIGH" and the "START - HOLD - DISCHARGE" switch should be at "START" with the Spark Chamber empty. The 556 High Voltage Power Supply should have already be turned on with the voltage set to zero volts and allowed to warm up for 5 minutes to reduce transients in the signal. Using the "COARSE" and "FINE" knobs, the voltage displayed on the High Voltage Power Supply meter should be adjusted to 1.20 kV. After the pre-sparking voltage has been set, the "START - HOLD - DISCHARGE" switch should be set to the "DISCHARGE" position to reduce the voltage to the Spark Chamber to zero Volts.

If the voltage displayed on the High Voltage Power Supply meter does not change as the knobs on the Voltage Controller are manipulated, the High Voltage Power Supply and the NIM Bin must be turned off and the Voltage Controller removed from the NIM Bin. The pins on the back of the Voltage Controller that connect it with the NIM Bin must be checked to ensure that they are firmly seated. The LF351N amplifiers in the Voltage Controller should be checked for damage and faulty connection with the circuit. The most common problems with the Voltage Controller have occurred with the connecting pins and with the improper seating of the LF351N amplifiers. However, if these checks do not correct the problem with the Voltage Controller, it is likely that the problem is due to a broken connection in the Voltage Controller or with an improper connection between the Voltage Controller and the 556 High Voltage Power Supply. After the problem has been detected and corrective measures have been taken, the Voltage Controller is carefully placed back into the NIM Bin, checking that the connecting pins come into full contact with their corresponding sockets in the NIM Bin. The procedure for setting the pre-sparking voltage can then be restarted.

After the switch on the Voltage Controller has been set to the "DISCHARGE" position, the display on the High Voltage Power Supply is allowed to return to zero volts before the "PULSE OUT" cable from the High Voltage Power Supply to the Spark Chamber Interface Circuit is disconnected. This removes the 590A Amplifier, the 770 Counter and the 771 Timer-Counter

34

from the circuit and protects them from high voltage signals generated during the pre-sparking process.

Using forceps or gloves, the Makrofol to be counted is removed from the sheet protector and carefully placed on the brass electrode in the Spark Chamber. The Makrofol should be placed such that the sample drop to be counted first is centered on the brass electrode and is as flat as possible on the electrode. A piece of the pre-cut Mylar should be selected and examined to determine if it is clean and free from rips and wrinkles. Once a suitable piece of Mylar has been found, it is examined to detect which side of the Mylar is aluminized. The aluminized side of the Mylar can be identified because it is much more reflective than the non-aluminized side.

The Mylar is placed in the Spark Chamber with the aluminized side of the Mylar in contact with the top of the Makrofol. The Mylar should completely cover the portion of the Makrofol on the electrode and is positioned with one end on the outer brass ring. The top of the Spark Chamber is then gently placed onto the brass guide posts and slid into place such that the rubber end of the central piston contacts the Mylar-Makrofol sandwich before the Plexiglas exterior is slid down so that it contacts the outer brass ring. Once the top of the Spark Chamber is in position, it is screwed into place using the rubber covered screws. Next a 100 gram brass weight is placed on top of the piston to insure good contact between the Mylar-Makrofol sandwich and the brass electrode.

After checking to verify that there are no foreign objects in contact with the Spark Chamber, it is now possible to proceed with the pre-sparking of the Makrofol. The "START - HOLD - DISCHARGE" switch is flipped to the "START" position to apply the pre-sparking voltage to the Spark Chamber and the digital timer is set to count down 1 minute. At the end of 1 minute, the "START - HOLD - DISCHARGE" switch is set to "DISCHARGE" and the Spark Chamber is allowed to completely discharge before the Spark Chamber is opened.

The Mylar is removed from the Makrofol using caution to not change the position of the Makrofol on the electrode. The Mylar is examined to verify that the entire sample drop was pre-sparked. This is verified by a circular pattern of holes in the Mylar that corresponds to the size

of the sample drop. Incompletely pre-sparked sample drops will result in a partial circular pattern of holes in the Mylar. If the sample drop has been completely pre-sparked, the Makrofol can be spark counted. Incompletely pre-sparked sample drops should be pre-sparked a second time after repositioning the Makrofol on the electrode such that the entire sample drop is on the surface of the electrode.

After the sample drop has been successfully pre-sparked, the black "Sharpie" pen can be used to mark an identifying code on the Mylar. The code should be of the following form: "R#(Rabbit number)-##(Makrofol number)-#(drop number)" with "Pre-#(number of the pre-sparking)" written below. For example, the code: "R26-32-A" with the designation "Pre-2" indicates that the Mylar is from the second pre-sparking of sample drop A from Makrofol 32 of Rabbit 26.

Before the Makrofol is spark counted, the spark counting voltage must be set on the Voltage Controller. During the process of setting the spark counting voltage, the Makrofol may remain in the Spark Chamber and the Makrofol, positioned on the electrode, as long as there is no Mylar in contact with the Spark Chamber. This allows the Makrofol to remain in the same position for spark counting and pre-sparking and does not harm the Makrofol, due to an incomplete circuit in the Spark Chamber when the Mylar is not on the Makrofol. To set the sparking voltage, the "HIGH - LOW" switch on the Voltage Controller should be set to "LOW" and the "START - HOLD - DISCHARGE" switch should be at "START". The 556 High Voltage Power Supply should be already on with the voltage set to zero volts. With the "COARSE" and "FINE" knobs, the voltage displayed on the High Voltage Power Supply meter is adjusted to 0.5 kV. After the spark counting voltage has been set, the "START - HOLD - DISCHARGE" switch is set to the "DISCHARGE" position to reduce to voltage to the Spark Chamber to zero Volts.

After the display on the High Voltage Power Supply reads zero volts, the "PULSE OUT" cable can be reconnected to the Spark Chamber Interface Circuit. Any data in the 771 Timer-Counter and the 770 Counter is cleared at this time.

A piece of the pre-cut Mylar is selected and examined for surface defects and contamination, and to determine which side is aluminized. The Mylar is placed in the Spark Chamber with the aluminized side of the Mylar in contact with the top of the Makrofol. The Mylar should completely cover the portion of the Makrofol on the electrode and is positioned with one end on the outer brass ring. The top of the Spark Chamber is gently placed onto the brass guide posts and slid into place such that the rubber end of the central piston contacts the Mylar-Makrofol sandwich before the Plexiglas exterior is slid down so that it contacts the outer brass ring. Once the top of the Spark Chamber is in position, it is screwed into place using the rubber covered screws. Next a 100 gram brass weight is placed on top of the piston to insure good contact between the Mylar-Makrofol sandwich and the brass electrode.

After checking to verify that there are no foreign objects in contact with the Spark Chamber, it is now possible to proceed with the spark counting of the Makrofol. The "START - HOLD - DISCHARGE" switch is flipped to the "START" position to apply the spark counting voltage to the Spark Chamber and the digital timer is set to count down 1 minute. At the end of 1 minute, the "START - HOLD - DISCHARGE" switch is set to "DISCHARGE" and the Spark Chamber is allowed to completely discharge before the Spark Chamber is opened.

The Mylar is removed from the Makrofol using caution to not change the position of the Makrofol on the electrode. The Mylar is examined to verify that the entire sample drop was spark counted. This can be verified by a circular pattern of holes in the Mylar that corresponds to the size of the sample drop. Incompletely spark counted sample drops will result in a partial circular pattern of holes in the Mylar. Incompletely spark counted sample drops should be spark counted a second time after repositioning the Makrofol on the electrode.

After the sample drop has been successfully spark counted, the black "Sharpie" pen is used to mark an identifying code on the Mylar. The code is of the following form: "R#(rabbit number)-##(Makrofol number)-#(drop number)" with "#(number of the spark counting)" written below. For example, the code: "R26-32-A" with the designation "3" indicates that the Mylar is from the third spark counting of sample drop A from Makrofol 32 of Rabbit 26.

The preceding steps for spark counting given above are repeated until the sample drop has been successfully spark counted 5 times. The Mylar used for pre-sparking and spark counting a sample drop on the Makrofol is stored together in a sheet protector. The sheet protector is then stored in a binder together with all the Mylar, Makrofol and data sheets from the same Rabbit.

Conclusion

The procedures given in this paper for the etching of fission tracks in Lexan and Makrofol have been used extensively in the study of environmental concentrations of ^{235}U and ^{239}Pu by the University of Utah's Environmental Radiation and Toxicology Laboratory. Fission tracks in Lexan and Makrofol have been successfully etched in more than 5000 samples using these procedures, with a negligible rate of failure.

In the past, environmental concentrations of ^{235}U and ^{239}Pu were studied using an alpha spectrometer. The ultimate detection limits of alpha spectrometry have been determined to be 0.01 pCi of ^{235}U and 0.01 pCi of ^{239}Pu. The detection limits of fission track analysis, as performed by the University of Utah's Environmental Radiation and Toxicology Laboratory using Lexan etched according to the procedures detailed in this paper, are 0.0005 aCi of ^{235}U and 20 aCi of ^{239}Pu. Further research is currently being performed to reduce these detection limits.

Fission tracks in Makrofol have also been successfully etched and counted in the Spark Chamber using the procedures given in this paper. Further research is also being performed on the Spark Chamber and its uses in the detection of environmental ^{235}U and ^{239}Pu [4].

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